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MULTIPLE DOPED ERBIUM LASER MATERIALS

Richard F. Woodcock

American Optical Corporation

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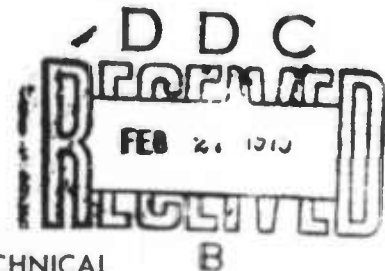
MULTIPLE DOPED ERBIUM  
LASER MATERIALS

FINAL REPORT  
by  
Richard F. Woodcock

January 1973

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DA Project No. 7910.21.702.55.01

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For

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Fort Monmouth, New Jersey

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## FOREWORD

The efforts reported herein were accomplished under Contract No. DAAB07-72-C-0053, Project No. 7910.21.702.55.01 for the Microwave and Quantum Electronics Branch of the Solid State and Frequency Control Division of the Electronics Components Laboratory, U.S. Army Electronics Command, Fort Monmouth, New Jersey. Contractors representative were Dr. E. Schiel and Dr. H. Hieslmair.

The work was carried out by the Basic Materials Research Department of the Research Division, American Optical Corporation in Southbridge, Massachusetts under the direction of Dr. Richard F. Woodcock who was project scientist and author of this report.

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## MULTIPLE DOPED ERBIUM LASER MATERIALS

### 1. INTRODUCTION

The goal of this work is to develop an erbium doped glass laser whose performance characteristics will exceed those of existing erbium laser materials with an ultimate goal of achieving a 50 millijoule output for an input of 50 joules or less.

In previous work<sup>1,2</sup> both silicate-type glass and phosphate-type glass were investigated as host media for homogeneous erbium laser rods, and clad erbium laser rods of the silicate-based glasses were investigated in which the sensitizing agent was incorporated in the cladding material.

The homogeneously doped phosphate materials have better laser performance characteristics than do the homogeneously doped silicate-based materials. This was attributed to a higher oscillator strength of the phosphate-based materials. These materials have the disadvantage that the yield of good optical quality glass is lower and difficulties have been encountered in obtaining high purity raw ingredients and high purity crucibles for the manufacture of iron-free phosphate laser materials. In the previous phosphate studies, encouraging results were obtained when single crystal grade ammonium dihydrogen phosphate (ADP) was used as a source of high purity phosphate raw material. A continuation of the investigation of the use of high purity raw ingredients and high purity crucibles for the fabrication of high purity phosphate materials was to be continued.

The initial studies indicated that  $\text{Nd}^{3+}$ -ions and  $\text{Yb}^{3+}$ -ions serve as sensitizing agents for the active  $\text{Er}^{3+}$ -ion. The  $\text{Yb}^{3+}$ -ions also serve as agents to transfer energy from excited sensitizer ions to the  $\text{Er}^{3+}$ -ions. Unfortunately, the  $\text{Nd}^{3+}$ -ion also tends to quench neighboring  $\text{Er}^{3+}$ -ions in an excited state and provides a small but finite amount of absorption at the emission wavelength due to an absorption band centered at about  $1.58 \mu\text{m}$ .

The goal of the clad configuration was to isolate the sensitizing agents in a non-lasing cladding so that its advantages are still utilized and its disadvantages are eliminated. The

studies on clad rods were carried out in silicate-based materials because it is easier to make good optical quality silicates and the laser properties of the silicate glasses are more reproducible from one melt to the next, thus minimizing the possibility of spurious results due to variations in the host material. Results of these studies indicate that the clad silicate-based laser rods have better laser performance characteristics than unclad phosphate-based laser rods.

The intent of the present work is to develop a clad phosphate-based erbium laser rod which would have the combined advantages of the clad laser rod configuration and the higher oscillator strength of the phosphate-based materials. The general approach of the present effort is to optimize the parameters of clad laser rods using silicate-based glass and to develop a high purity clad phosphate laser rod configuration. The silicate material was used to optimize the rod configuration because some clad material already exists with "standard cavity" data and because the silicates are reliable materials to work with.

The clad phosphate program was scheduled to include, (1), a continuation of the work previously started to fabricate high purity phosphate laser materials, (2), an investigation of the compatibility and "drawing" properties of phosphate materials required for the fabrication of clad phosphate laser rods (with possible modification of the core and cladding compositions if incompatibilities were found to exist), (3), an investigation of other possible sensitizing agents in addition to  $\text{Nd}^{3+}$ -ions and  $\text{Yb}^{3+}$ -ions to be included in the cladding glass, and (4), the evaluation of an optimized clad erbium phosphate laser rod.

The major problem encountered in this program was the production of high purity phosphate laser materials due to crucible failure associated with the form of high purity raw ingredients being used. A solution to this problem was obtained, but too late in the program for the fabrication of optimized phosphate clad laser rods, and their subsequent evaluation. Evaluation has been completed for unoptimized clad phosphate rods made from materials which were produced earlier. These laser rods satisfied the ultimate contract goal of 50 mJ output at a 50 J input thus demonstrating the feasibility of this approach.

## 2. LASER ROD CONFIGURATIONS

### 2.1 IMPROVED "DOUBLE-U" DESIGN

The conventional clad laser rod configuration is that of a cylindrical core of active laser glass clad with a concentric cylinder of sensitized cladding glass. When the ultimate in laser efficiency is required, a "double-U" laser rod-flashlamp configuration, as shown in Figure 1a, is sometimes used. The advantage of this configuration is the better optical coupling of the flashlamps which fit snugly into the semicylindrical grooves running the length of the laser rod. Good results were obtained with an erbium laser rod of this configuration where the core glass contained  $\text{Er}_2\text{O}_3$  and  $\text{Yb}_2\text{O}_3$  and the cladding glass was uniformly doped with  $\text{Nd}_2\text{O}_3$  and  $\text{Yb}_2\text{O}_3$ . Although this system works better than the standard cylindrical configuration, probably only a small percentage of the fluorescent light originating in the sharp corners of the cladding glass ever reaches the core glass.

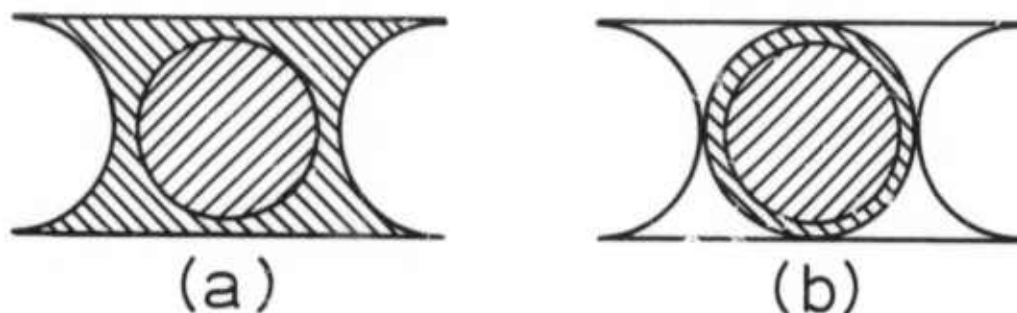


Figure 1. Cross Section of Double-U Laser Rod Configurations  
(a) Active cylindrical core with "double-U" cladding uniformly doped with  $\text{Nd}_2\text{O}_3$  and  $\text{Yb}_2\text{O}_3$  to provide both sensitizing cladding and optical coupling.  
(b) Active cylindrical core with cylindrical cladding doped with  $\text{Nd}_2\text{O}_3$  and  $\text{Yb}_2\text{O}_3$  to provide sensitization and an undoped "double-U" cladding to provide optical coupling.

A laser rod configuration which may utilize more completely the combined advantages of improved optical coupling provided by the "double-U" configuration and improved pumping provided by a sensitized cladding, is shown in Figure 1b. In this configuration, the active core glass containing  $\text{Er}_2\text{O}_3$  and  $\text{Yb}_2\text{O}_3$  is surrounded by an intermediate cylindrical sensitized cladding containing  $\text{Nd}_2\text{O}_3$  and  $\text{Yb}_2\text{O}_3$  ions, and this composite in turn is imbedded in a "double-U" configuration of clear glass.

In order to make a comparison between the two configurations in Figure 1 it would be desirable to have the core glass and sensitized cladding glass of these two rods made from the same glass composition, preferably from the same melts of these compositions. A small amount of the latter was located in existing stock in the form of clad cylindrical rods with a 4 mm core and an 8 mm cladding. The sensitized cladding of this stock was ground down to a 5.3 mm OD for subsequent fabrication of a "double-U" rod with a 3 mm core, a 4 mm intermediate sensitized cladding, and a clear outer cladding. Strain measurements made on samples of the core glass fused to various clear glasses indicate that a 1045 ophthalmic crown glass would be a very suitable material for the clear cladding component of this composite laser rod configuration.

The 5.3 mm OD cylindrical rod, to be used as the core and intermediate cladding of the improved double-U configuration, was placed inside a rectangular "tube" of 1045 crown, (a bar of a rectangular cross section with a cylindrical bore) and drawn down to the desired core diameter. This rectangular composite rod was subsequently ground to the desired "double-U" configuration. The core of the resulting laser rod was distorted in cross section and of poor optical quality due probably to the fabricating technique. The composite would normally have been made in a single draw from three separate pieces of larger size if sufficient raw stock had been available. Results on this rod were poor. A second attempt was not made due to the limited effort available under the contract.

## 2.2 OPTIMIZED CLAD CYLINDRICAL CONFIGURATION

The effect of the  $\text{Er}_2\text{O}_3$  concentration in the active laser core on laser performance was reported previously.<sup>2</sup> A summary of these results is given in Table I. A standard cavity was used in that study which consisted of a clad laser rod with a 4 mm core diameter, 8 mm cladding diameter, and 76 mm length which was pumped by two xenon filled flashlamps with 4 mm bore and 76 mm arc length, close wrapped with silver foil, output reflectivities of 65 or 80%, a 4,460  $\mu\text{H}$  inductor and a 760  $\mu\text{F}$  capacitor. As

TABLE I. Effect of  $\text{Er}^{3+}$ -Ion Content on Laser Performance

Rod Composition		Nominal $\text{Er}_2\text{O}_3$ conc. (wt%) <sup>a</sup>	Output Reflector (%)	Laser Threshold (J) <sup>b</sup>	Slope Efficiency (%)	Input for 50 mJ Output (J)
Core	Cladding					
MG-2110A	MG-2400B	1.0	55 80	350 325	0.33 0.2	365 360
MG-2499	MG-2400C	0.7	65 80	237 225	0.55 0.43	235 235
MG-2495	MG-2400B	0.5	65 80	165 165	0.47 0.36	180 180
MG-2453A	MG-2400B	0.25	65 80	150 145	0.37 0.29	160 160
MG-2450A	MG-2400B	0.20	65 80	125 120	0.16 0.20	160 145
MG-2430A	MG-2400B	0.15	65 80	125 95	0.08 0.09	185 150
MG-2445A	MG-2400B	0.10	65 80	200 125	0.04 0.05	305 225

<sup>a</sup>Core diameter 4 mm, cladding diameter 8 mm, rod length 76 mm.<sup>b</sup>Two xenon-filled flashlamps 4 mm bore  $\times$  76 mm arc length, close-wrapped with Ag-foil, 4460  $\mu\text{H}$  inductor, and 760  $\mu\text{F}$  capacitor.

indicated in the table, best efficiency for a 50 mJ output was obtained at rather low  $\text{Er}_2\text{O}_3$  concentrations. At that time a preliminary study of the effect of laser rod length on laser performance was made which indicated that the best overall efficiency for a 50 mJ output, for a given core and cladding combination, was obtained with a fairly short rod length.<sup>2</sup> In that experiment the total  $\text{Er}_2\text{O}_3$  concentration was fixed, thus the total  $\text{Er}^{3+}$ -ion content of the rod was decreased as the rod length was decreased and it was not clear whether the improvement was due to the shorter length or the lower  $\text{Er}^{3+}$ -ion content.

In the present study an attempt was made to determine the effect on laser performance of the individual laser parameters. The effect of changing laser rod length on laser performance was carried out using a variety of  $\text{Er}_2\text{O}_3$  concentrations such that the total  $\text{Er}^{3+}$ -ion content of the active core remained approximately constant. In all cases the core diameter was 4 mm and the flashlamps had a 3 mm bore with arc length equal to that of the laser rod being tested. Results of this study, shown in Table II, indicate that best performance is obtained with the shorter laser rod length and thus the previous results were not due solely to a decrease in total  $\text{Er}^{3+}$ -ion content.

A similar study was carried out to investigate the effect of laser rod diameter on laser performance. In this case the rod length was held constant at 64 mm and the  $\text{Er}_2\text{O}_3$  concentration was chosen such that the total  $\text{Er}^{3+}$ -ion content of the active core remained approximately constant. Results of this study, given in Table III, indicate that the best laser performance is obtained when the diameter of the active core is equal to or slightly greater than the bore diameter of the flashlamps. As one might expect, the optimum reflectivity of the output mirror increases as the  $\text{Er}_2\text{O}_3$  concentration, (and thus the gain of the rod) decreases.

These results would suggest that the optimum laser rod configuration for a 50 mJ output would be one with an  $\text{Er}_2\text{O}_3$  concentration of about 0.2 wt%, a core diameter which matches the bore diameter of the flashlamps (3 mm in the present case), and a length of 38 mm. With this number of ions per unit length, and the short rod length, the gain of the rod would probably require a rather high output mirror reflectivity and thus the losses within the rod may play a predominant role in cavity optimization. Final optimization of rod dimensions could probably be achieved by investigating rods with  $\text{Er}_2\text{O}_3$  concentrations of 0.20 to 0.25 wt%, a core diameter of 3 mm, and various rod lengths from 38 to 64 mm. Time was not available for the preparation of the laser rods required for this final step in optimization.



TABLE II. Effect of Laser Rod Length on Laser Performance

Rod Composition		Nominal Er <sub>2</sub> O <sub>3</sub> conc. (wt%)	Rod Length <sup>a</sup> (mm)	Output Reflector (%)	Laser Threshold <sup>b</sup> (J)	Slope Efficiency (%)	Input for 50 mJ Output (J)
Core	Cladding						
MG-2101	MG-2400	0.3	38	65	77	0.22	98
				80	68	0.23	90
				90	80	0.09	137
MG-2453	MG-2400	0.25	50	65	92	0.26	106
				80	93	0.23	116
				90	90	0.11	136
MG-2450	MG-2400	0.20	64	65	92	0.35	104
				80	100	0.34	112
				90	100	0.17	124
MG-2430	MG-2400	0.15	76	65	100	0.15	130
				80	92	0.13	130
				90	148	0.08	148
MG-2445	MG-2400	0.10	114	65	100	0.06	173
				80	104	0.19	128
				90	150	0.06	232

<sup>a</sup>All laser rods have 4 mm core diameter (6 mm cladding diameter) therefore total Er<sup>3+</sup>-ion content approximately constant.

<sup>b</sup>Two xenon-filled flashlamps 3 mm bore with arc length equal rod length, close-wrapped with Ag-foil, 4460  $\mu$ H inductor, 760  $\mu$ F capacitor.



TABLE III. Effect of Laser Rod Diameter on Laser Performance

Core	Cladding	Nominal $\text{Er}_2\text{O}_3$ conc. (wt%)	Rod Core Diameter <sup>a</sup> (mm)	Output Reflector (%)	Laser Threshold <sup>b</sup> (J)	Slope Efficiency (%)	Input for 50 mJ Output (J)
MG-2449	MG-2400	0.7	2	65 74.5 80 90	108 112 112 100	0.23 0.18 0.17 0.06	125 135 135 167
MG-2101	MG-2500 <sup>c</sup>	0.3	3	65 74.5 80 90	70 62 85 100	0.11 0.10 0.10 0.06	110 100 125 167
MG-2450	MG-2400	0.2	4	65 74.5 80 90	110 95 90 87	0.24 0.25 0.26 0.11	130 115 110 135
MG-2445	MG-2400	0.1	6	80 90 95	170 118 118	0.14 0.10 0.08	200 160 170

<sup>a</sup>All rods were 64 mm long therefore total  $\text{Er}^{3+}$ -ion content approximately constant.

<sup>b</sup>Two xenon-filled flashlamps 3 mm bore and 64 mm arc length, close-wrapped with Ag-foil, 4460  $\mu\text{H}$  inductor, and 760  $\mu\text{F}$  capacitor.

<sup>c</sup>Similar to MG-2400 but contains 3 wt%  $\text{Yb}_2\text{O}_3$  rather than 4 wt%.

### 3. CLAD PHOSPHATE LASER RODS

#### 3.1 SENSITIZED PHOSPHATE CLADDING GLASS

The main reason for going to a clad laser rod configuration was to preserve the advantages of using  $\text{Nd}^{3+}$ -ions as a sensitizing agent while eliminating their disadvantages. This technique could also be applied to other possible sensitizing agents which were not previously considered for various reasons. An investigation of other sensitizing agents for use in the cladding of silicate-based clad laser rods has previously been reported.<sup>2</sup> The phosphate-based material provides a markedly different host for the sensitizing ions than does the silicate-based glass, and therefore the various sensitizing agent candidates need to be reconsidered.

A series of small (50 gm) melts of zinc-aluminum-phosphate glasses was made which contained 4 wt%  $\text{Yb}_2\text{O}_3$ . In most cases these glasses also contained 4 wt%  $\text{Nd}_2\text{O}_3$ . An additional sensitizing agent candidate was also present in most of these glasses, namely 1-4 wt%  $\text{CeO}_2$ , 0.1-6 wt%  $\text{MnO}$ , 0.2-1.0 wt%  $\text{Cr}_2\text{O}_3$ , 0.2-1.0 wt%  $\text{UO}_2$ , 0.5-4.0 wt%  $\text{MoO}_3$ ,  $\text{PbO}$  or  $\text{Sb}_2\text{O}_3$ . The  $\text{CeO}_2$  was frequently added in combination with the latter oxides.

One step in the sensitizing process of clad rods is the fluorescent emission from  $\text{Yb}^{3+}$ -ions in the cladding glass which is subsequently absorbed by the  $\text{Yb}^{3+}$ -ions in the core glass. Thus, a rough indication of the effectiveness of the various sensitizing agents in the cladding glass may be obtained by monitoring the  $\text{Yb}^{3+}$ -ion fluorescence from these glasses. Three different types of sample irradiation were employed. The first consisted of an unfiltered 100 W high pressure mercury arc lamp with quartz optics. Fluorescent emission in this case gives an indication of the combined effects of sensitizer excitation and direct  $\text{Yb}^{3+}$ -ion excitation. For those samples which showed significant  $\text{Yb}^{3+}$ -ion emission in this case, a "heat screen" filter was added to the light source which removes the UV and  $\text{Yb}$ -ion pump bands from the excitation spectrum. This gives an indication of the efficiency of the sensitizer ions which are excited by the visible region of the spectrum. These samples are then also pumped through a narrow bandpass filter which transmits light centered at 970 nm to excite just the  $\text{Yb}^{3+}$ -ions. The latter is used as a monitor for possible variations in the  $\text{Yb}^{3+}$ -ion concentration in the glass.

Excitation in all cases is incident upon the surface of sample at an angle of about  $45^\circ$  and emission is observed normal to this same surface to minimize reabsorption of the emitted

light in the 970 nm region. Any error in emission intensity due to scattering of source light from the sample, particularly at the 1.014  $\mu\text{m}$  mercury line, is accounted for by observing the mercury line at 1.1  $\mu\text{m}$ . The 880 nm line was also monitored to detect fluorescent emission from the  $\text{Nd}^{3+}$ -ion rather than the transfer from the  $\text{Nd}^{3+}$  to the  $\text{Yb}^{3+}$ -ion. In all cases the 800 nm emission was essentially zero. Some of these results are tabulated in Table IV. Yb fluorescent emission was measured at 970, 1010, and 1050 nm, but only the 1010 nm, assumed to be the most accurate value, is reported here. Also included in this table are the fluorescent lifetimes of the  $\text{Yb}^{3+}$ -ion to give an indication of quenching of the excited  $\text{Yb}^{3+}$ -ion by the sensitizing agent. Where lifetime data is omitted, no measurement was made because the intensity of fluorescent emission was too low, or the lifetime was too short to measure accurately (except in the case of more recent glasses MG-2806, 2807 and 2809).

Absorption measurements were also made on the samples to give an indication of the valence state of the sensitizer ion in the glass. The intensity of the Yb absorption band corroborates the emission data which indicates that the  $\text{Yb}_2\text{O}_3$  concentration in the glass is essentially constant within measuring limits of error.

As expected, the most dramatic improvement in  $\text{Yb}^{3+}$  fluorescence is obtained by the addition of  $\text{Nd}^{3+}$ -ions to the glass. Preliminary results indicate that the addition of  $\text{Ce}^{3+}$ -ions or  $\text{Pb}^{2+}$ -ions, added singly to the  $\text{Yb}^{3+}$  glass, provides some sensitization but that  $\text{Ce}^{3+}$  or  $\text{Pb}^{2+}$  in combination with  $\text{Nd}^{3+}$  is about the same as  $\text{Nd}^{3+}$  alone.

For the samples containing MnO as the sensitizing agent, the melts containing 2 wt% CeO and only 1 wt% sugar to provide a reducing condition within the melt, all have very low  $\text{Yb}^{3+}$ -ion emission and the transmission curve indicates varying degrees of absorption throughout the visible region due to the presence of  $\text{Mn}^{3+}$ -ions. MG-2782 and MG-2783 were subsequently melted with the sugar content of the batch composition increased to 2 wt%. Transmission spectra of these two melts indicate that the Mn is present as the  $\text{Mn}^{2+}$ -ion but total Mn absorption is rather low due to the fact that the extinction coefficient of the  $\text{Mn}^{2+}$ -ion is about two orders of magnitude less than that of the  $\text{Mn}^{3+}$ -ion.<sup>4</sup> Subsequent melts (MG-2806, MG-2807 and MG-2809) were made with increased concentrations of MnO, with 1 wt% sugar added, and with the cerium removed or replaced by arsenic. These glasses showed an enhanced  $\text{Mn}^{2+}$ -ion concentration but also had color streaks and transmission spectra which indicated the presence of varying amounts of  $\text{Mn}^{3+}$  also. These results suggest that the

TABLE IV. Relative Efficiency of Sensitizing Agents

Melt Number	Sensitizing Agent (wt% of oxide)										Yb <sup>3+</sup> Emission at 1.01 μm (arb. units)			
	Nd	Yb	Ce	Mn	Cr	U	Mo	Pb	Sb	As	unfiltered excitation	visible excitation	0.970 μm excitation	τ <sub>Yb</sub> (ms)
MG-2747D	4										.6	-	.4	-
MG-2748B	4										4.8	2.6	.3	1.02
MG-2755A	4		2								1.3	-	-	-
MG-2756A	4		1								4.5	2.5	.3	1.06
MG-2757A	4		2								4.2	2.1	.35	1.13
MG-2758A	4		4								3.9	2.0	.35	1.10
MG-2759A <sup>a</sup>	4			1							0	-	-	-
MG-2760A <sup>a</sup>	4		2	0.5							0.7	-	-	0.32
MG-2761A <sup>a</sup>	4		2	1							0	-	-	-
MG-2762A <sup>a</sup>	4		2	4							0	-	-	-
MG-2782A <sup>b</sup>	4		2	0.1							5.9	2.5	.4	1.3
MG-2783A <sup>b</sup>	4		2	0.5							5.4	2.7	.35	1.26
MG-2806A <sup>a</sup>	4		2	6							2.0	0.8	.4	-
MG-2807A <sup>a</sup>	4		2	2.5							2.5	1.1	.4	-
MG-2809A <sup>a</sup>	4		2	2						2	1.2	0.5	-	-
MG-2763A <sup>a</sup>	4				0.5						0	-	-	-
MG-2764A	4		2		0.2						0.6	-	-	0.37
MG-2765A <sup>a</sup>	4		2		0.5						0	-	-	-
MG-2766A <sup>a</sup>	4				1						0	-	-	-
MG-2768A	4					0.5					0	-	-	-
MG-2769A	4		2		0.2						0.5	-	-	-
MG-2770A	4		2		0.5						~0	-	-	-
MG-2771A	4		2		1						~0	-	-	-
MG-2772A <sup>a</sup>	4						1				0.1	-	-	-
MG-2773A <sup>a</sup>	4		2				0.5				0.5	-	-	-
MG-2775A	4		2				1				0	-	-	-
MG-2777A	4							5			1.2	-	-	-
MG-2778A	4							5			3.8	2.2	.3	1.18
MG-2780A	4								1		0	-	-	-
MG-2781A	4								1		0.5	-	-	1.0

<sup>a</sup> 1 wt% sugar added<sup>b</sup> 2 wt% sugar added to increase reducing conditions of the melt

latter melts should be remade with a higher concentration of sugar added to the batch or otherwise increase the reducing conditions of the melt. Fluorescent lifetime measurements were not made on the last three glasses but results on the other glasses containing MnO indicate that the presence of  $Mn^{3+}$ -ions tends to quench the emission from excited  $Yb^{3+}$ -ions.

The addition of  $Cr_2O_3$  to the cladding glass composition resulted in the formation of the desired  $Cr^{3+}$ -ions but produced very poor  $Yb^{3+}$  fluorescent intensities.<sup>5</sup> The  $Cr^{3+}$  absorption bands were quite strong in all glasses indicating that the  $Cr_2O_3$  concentrations were probably too high. Fluorescent lifetime measurements of the  $Yb^{3+}$ -ion indicate that quenching is taking place due to the presence of  $Cr^{3+}$ -ions.

Several melts were made with varying concentrations of  $UO_2$  but absorption spectra indicate that the uranium is not present as the desired uranyl ion.<sup>6</sup> A subsequent melt, not included in Table IV, was made in which 2 wt%  $As_2O_3$  was substituted for the 2 wt%  $CeO$  in composition MG-2769A. This melt was made with oxygen bubbled through the glass during the melting process, in an attempt to drive the U to the  $UO_2^{2+}$  uranyl ion but the absorption spectra again indicates that the uranyl ion was not obtained.

Transmission spectra of the first two glasses of the series containing  $MoO_3$ , which were made with a reducing agent added to the batch, indicate that the Mo is present as the desired  $Mo^{3+}$ -ions totally or in part.<sup>6</sup> In both cases the  $Yb^{3+}$ -ion fluorescence appears to be severely quenched. Composition MG-2775 was made without the addition of a reducing agent to the batch composition and showed a strong absorption band at a slightly longer wavelength due presumably to the presence of  $Mo^{5+}$ -ions.

The glasses containing  $Sb_2O_3$  were strongly absorbing throughout the visible indicating a far too strong reducing condition.<sup>6</sup> Both glasses show considerable absorption at  $1.06 \mu m$  and very little fluorescence.

These results indicate that manganese shows the greatest promise of serving as a sensitizing agent in the cladding glass in combination with  $Nd^{3+}$  and  $Yb^{3+}$ -ions. The fabrication of clad rods with such a cladding glass was delayed until the problems associated with making phosphate glasses of high purity had been solved.

### 3.2 CORE AND CLADDING GLASS COMPATIBILITY

One of the first tasks in the development of a clad laser rod made from a phosphate-based glass is the determination of the compatibility of the core and cladding materials. In the silicate studies on clad configurations, the difference between the 15 wt% concentration of rare earth in the core and the 5 to 8 wt% concentration of rare earth in the cladding was compensated for by the addition of  $Y_2O_3$  to the cladding glass. A similar type of study has been carried out with the phosphate-based materials.

Compatibility of rheological properties cannot be determined from expansion data alone but must also take into consideration the strain or annealing temperature of the glass. Compatibility is therefore determined by a "strain-fusion" measurement. The test sample for this measurement is prepared by fusing together, in sandwich fashion, a 10 mm x 10 mm x 30 mm bar of core material between two 3 mm x 10 mm x 30 mm plates of cladding material. The 16 mm x 30 mm surfaces, normal to the interfaces between the core and cladding glasses, are then polished and the resulting stress birefringence induced at the interfaces is measured over the 10 mm path along the interface.

Results of some of these measurements are summarized in Table V and the glass compositions are given in Table VI. Values given are the average values of retardation (nm/cm) due to tension in the cladding (negative values represent compression). Measurements were made on a series of cladding glass containing varying amounts (5-10%) of  $Y_2O_3$  fused to a core glass containing 15%  $Yb_2O_3$  and 0.5%  $Er_2O_3$ . The best results in this series of phosphate glasses were obtained when 9 to 10 wt%  $Y_2O_3$  (MG-2416 and MG-2419) was present in the cladding glass. The strain-fusion data indicated that tension in the cladding glass decreases with increased  $Y_2O_3$  concentration and that additional  $Y_2O_3$  would be required to reach the point where the cladding would no longer be in tension. The initial melts of this series indicated that there may be problems with devitrification of the compositions containing concentrations of 8 wt%  $Y_2O_3$  and higher.

To circumvent this problem, additional strain-fusion measurements have been made between cladding glasses MG-2416 and MG-2419 and other core compositions containing approximately 15 wt%  $Yb_2O_3$  in which the host composition has been modified. Replacement of 2 wt% of  $Al_2O_3$  by  $B_2O_3$  and 1% of  $P_2O_5$  by  $SiO_2$  (MG-2444 and MG-2460) reduces the tension in the cladding dramatically when fused with MG-2416 and puts the cladding into compression when fused with MG-2419. This shift towards compressive

TABLE V. Rheological Compatibility of Core and Cladding

"Cladding"	"Core"	Average Tensile Stress in Cladding ( nm/cm)
<u>( Increasing <math>Y_2O_3</math> in Cladding)</u>		
2405	2403	292 ( sample cracked)
2406	2403	296
2411	2403	242
2404	2403	179 ( sample cracked)
2416	2403	163
2419	2403	117
<u>( Substituting <math>B_2O_3</math> for <math>Al_2O_3</math> and <math>SiO_2</math> for <math>P_2O_5</math> in core)</u>		
2419	2444	- 67
2416	2460	64
2152	2444	-175
<u>( Decreasing <math>Al_2O_3</math> in Core)</u>		
2416	2133	595 ( sample cracked)
2416	2320	150
2416	2329	- 55
2416	2330	-304
<u>( Increasing <math>Al_2O_3</math> in Cladding)</u>		
2767	2460	-180
<u>( Substituting MgO for ZnO)</u>		
2416	2238	190 ( Sample cracked)



TABLE VI. Composition of Glasses in Table V

Melt Number	Composition (wt%)									
	ZnO	MgO	Al <sub>2</sub> O <sub>3</sub>	B <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	Nd <sub>2</sub> O <sub>3</sub>	Yb <sub>2</sub> O <sub>3</sub>	Er <sub>2</sub> O <sub>3</sub>	Y <sub>2</sub> O <sub>3</sub>
(Cladding)										
2405	15.5		8.9			64.6	3	3		5
2406	15.3		8.9			63.9	3	3		6
2411	15.1		8.7			63.2	3	3		7
2404	14.9		8.6			62.5	3	3		8
2416	14.8		8.5			61.7	3	3		9
2419	14.6		8.4			61.0	3	3		10
2152	14.7		8.4			61.3	0.2	14.9	0.5	
2767	13.1		10.7			63.2	3	3		7
(Core)										
2403	14.7		8.4			61.4		15	0.5	
2444	14.7		6.4	2	1	60.3	0.2	14.9	0.5	
2460	14.7		6.4	2	1	60.3		15.3	0.3	
2133	8.4		14.7			61.3	0.1	15	0.5	
2320	11.4		3.3			62.5	0.2	14.9	0.5	7.2
2329	11.4		1.3			68.5	0.2	14.9	0.5	3.2
2330	11.4					73.0	0.2	14.9	0.5	
2238		8.0	9.2			67.2	0.2	14.9	0.5	



stress in the cladding was verified by fusing MG-2152 and MG-2444 together where the only difference in composition is the presence of  $B_2O_3$  and  $SiO_2$  in the latter glass.

Substituting MgO (MG-2238) for ZnO in an MG-2403-type core glass appears to shift the tension in the wrong direction. Replacing half of the  $Al_2O_3$  content of an MG-2403-type core glass by  $Y_2O_3$  (MG-2320) on a molar basis has little effect on the cladding tension (152 nm/cm vs 163 nm/cm). Decreasing the combined concentration of  $Al_2O_3$  and  $Y_2O_3$  by one half or eliminating these ingredients completely, MG-2329 and MG-2330 respectively, dramatically switches the cladding glass into compression thus indicating that the improvement observed in the MG-2444 and MG-2460 fusions was probably due to a decrease in alumina rather than the presence of  $B_2O_3$  or  $SiO_2$ .

The MG-2416 and MG-2419 cladding compositions in combination with the MG-2444-type glasses containing  $B_2O_3$  and  $SiO_2$  have an acceptable degree of compatibility. However, the cladding glasses as indicated above may be difficult to fabricate without the presence of devitrification. The combinations in which the  $Al_2O_3$  (plus  $Y_2O_3$ ) content of the active core glass was reduced suggests that acceptable levels of compatibility can be reached with these core glasses, however, the lasing properties of these materials as core glasses has not been established. Materials such as MG-2444 have demonstrated that they have good laser characteristics. Therefore, a cladding glass, MG-2767, was fabricated in which the  $Al_2O_3$  concentration of an MG-2411-type glass was increased by 2 wt% in an effort to provide an acceptable cladding glass for use in combination with core glasses of good laser performance. Strain-fusion results were good. However, the glass appeared to contain some undissolved ingredients suggesting that the  $Al_2O_3$  concentration may be too high. It should be possible, however, to arrive at a concentration which provides compatibility with a core material and it is possible to fabricate good optical quality glass.

Attempts to make high purity melts of the MG-2329 and MG-2330 compositions to check their laser properties were unsuccessful due to crucible failure. Subsequent melts of MG-2416 and MG-2419 have been satisfactorily made without devitrification problems. It would therefore appear that there are several possibilities for core and cladding glass combinations which are compatible rheologically.

Some care was required in interpreting the strain-fusion results because cracking occurred in some of the samples which may have tended to release the stress in the sample. In these cases

the values given in Table V may be too low. In some cases, one or both components of the samples discolored during fusion indicating that the sample was probably no longer a single-phase, glassy system. These data were discarded since the material had been altered.

In the fabrication of clad rods, the core and cladding glasses are simultaneously fused and drawn to a predetermined diameter. In light of the devitrification problems with the initial glass melts, studies were initiated to gain some knowledge of the drawing characteristics of phosphate glasses. These studies were carried out using phosphate glasses made from ingredients of questionable purity but which are easily made into glass. In this way, the problems encountered in melting high purity phosphate glasses did not hold up the whole program.

Initial drawing experiments were carried out with just the cladding glass MG-2416 containing 9 wt%  $Y_2O_3$ , the initial melts of which indicated some potential problems with devitrification (cf. Section 3.2). The drawing operation was performed over a range of furnace temperatures between 1540°F and 1400°F. The actual temperature of the glass moving through the furnaces is somewhat less than this but it is not easily monitored. The upper temperature is that at which the glass can be "bailed," i.e. gripped with tongs and drawn manually. The lower temperature is that at which a rod about 2 mm in diameter, being machine drawn at a slow uniform rate, breaks under tension. Between these two limits rods were drawn successfully which showed only a few signs of surface devitrification.

This result was very encouraging since it indicates that it may be possible to draw phosphate rods without modifying the present glass compositions. Based on this experience, clad phosphate rods were drawn using the MG-2416 glass as a cladding and MG-2146 as the core material. The latter is a zinc aluminophosphate glass, similar to the cladding glass composition but containing 15 wt%  $Yb_2O_3$  and 0.5 wt%  $Er_2O_3$ . Rods prepared from this clad stock were of very poor optical quality, but appeared to be free of devitrification or discoloration as observed in the strain-fusion samples. The latter when it exists is the result of heating just enough to cause phase separation, a precursor to devitrification. This limited experience indicates that drawing conditions should be achievable which will permit the fabrication of the desired clad phosphate rods once the pure materials are available.

### 3.3 HIGH PURITY PHOSPHATE CORE AND CLADDING GLASSES

The major problem encountered in homogeneously doped phosphate materials<sup>1</sup> was the variation in laser performance from one melt to the next which was attributed to impurities in the raw ingredients. One of the primary efforts of the present program was to be the development of ultrahigh purity phosphate laser materials. This requires a source of high purity raw ingredients from which the glass batch can be mixed and a crucible of high purity in which the glass can be melted. Locating a source of pure phosphate material has been a prime concern from the beginning of the program since  $P_2O_5$  is the major constituent of the glass. The phosphorous may be in the form of the oxide or any other compound as long as it will not alter the final glass composition. Pure sources of this material amenable to the glass melting process have not proven to be readily available.

The purest source of  $P_2O_5$  obtained to date is crystalline ammonium dihydrogen phosphate (ADP). Difficulties were encountered with crucible failure when attempts were made to melt glasses in high purity alumina crucibles using this material. Conversely, no melting problems were encountered when glasses with  $Al(PO_3)_3$  as the major ingredient are melted in "porcelain" crucibles. These glasses would be unsuitable for the present purpose because both the  $Al(PO_3)_3$  and the porcelain crucibles are known to contain large amounts of impurities. Attempts to isolate the source of this problem evolved into the following subtasks: (1) An investigation of a crucible material which would be suitable for use with the high purity ADP; (2) an investigation of methods of obtaining purer  $Al(PO_3)_3$ ; (3) altering the time-temperature cycles to make it possible to use ADP with the present high purity crucibles.

Attempts to make glass using  $Al(PO_3)_3$  in high purity crucibles have been successful, while attempts to melt glass using ADP in porcelain crucibles were unsuccessful. These results indicate that the problem is probably associated with thermal shock of the crucible due to the water vapor given off by the ADP as it decomposes with increasing temperature. The temperatures at which ADP melts, gives off ammonia and gives off water vapor have been determined. Attempts to alter the heating cycle such that the water may be driven off at a lower temperature followed by a normal melting cycle did not lead to a satisfactory glass melting procedure.

Efforts to form a high purity  $Al(PO_3)_3$  by prereacting ADP with a high purity aluminum compound were not very successful.  $AlPO_4$  was successfully synthesized in this manner, but this compound requires that additional  $P_2O_5$  in some form be added to the

batch composition if the composition of the final glass is to be preserved. A melt was attempted using this prereacted high purity  $\text{AlPO}_4$  with high purity ADP, but there still appeared to be enough water present in the ADP to cause crucible failure.

An alternate method of utilizing the existing  $\text{Al}(\text{PO}_3)_3$  as a starting ingredient is to carry out the melting process under highly oxidizing conditions in an attempt to drive all of the available iron into the  $\text{Fe}^{3+}$  valence. Glasses were melted with oxygen bubbled through the glass during the melting process plus the addition of a variety of oxidizing agents to the batch composition in an attempt to do this. Absorption at the center of the  $\text{Fe}^{2+}$ -ion band was reduced from about 11% per cm to about 2% per cm when  $\text{As}_2\text{O}_3$  was used as an oxidizing agent in the glass. Improvement, but of a lesser degree, was also obtained with the addition of  $\text{Sb}_2\text{O}_3$ ,  $\text{MnO}$  and  $\text{CeO}_2$ . Although the results were encouraging, this approach by itself still leaves an undesirable concentration of  $\text{Fe}^{2+}$ -ions present in the glass.

A variety of different alumina, mullite and porcelain crucible types were investigated in an attempt to find a suitable material for use with ADP. Unfortunately, as the purity of the crucible is increased, the thermal expansion coefficient tends to increase and the susceptibility to thermal shock becomes worse. Attempts to use pure crucible materials with a more porous structure to improve thermal shock resistance did not solve the fracture problem.

Crucibles were ordered of high purity fused silica which nevertheless have considerably higher iron content than either pure alumina or pure mullite crucibles. Successful melts using ADP were made in these crucibles but they showed a higher degree of crucible attack than either mullite or alumina. One would therefore expect a considerably higher level of contamination in melts made from these crucibles. This is one possible solution to the crucible problem but puts restrictions on the maximum melting temperature and melt time that can be used and on the "purity" of the final product.

An alternate solution to the problem is to form a hole in the bottom of the fused silica crucible and use this as a premelting stage above a conventional flat bottomed mullite or alumina crucible. In this manner, the phase of the melting cycle in which water vapor is released is carried out in a silica crucible which has low thermal expansion and thus, high thermal shock resistance. In the molten state the glass immediately flows through the orifice of this crucible into the high purity crucible below for the fining and homogenization phase of the

process. Thus the introduction of impurity ingredients from the fused silica crucible is kept to a minimum due to a short dwell time in the premelter. Glasses using ADP were successfully melted in this manner which further indicates that the problem is one of thermal shock rather than some other mechanism such as attack at the grain boundaries with subsequent weakening and rupture of the wall.

Crucibles of high purity alumina and mullite were also ordered with a variety of wall thicknesses and a rounded bottom to relieve the stress concentrations which were felt might exist at the "heel" of the conventional flat bottomed crucibles. Unfortunately, a rather long delivery time was required for fabrication of these special crucibles and the crucibles were not received until late in the contract period. Several melts have been made with these crucibles, and to date, none have failed in the melting process.

Thus, although the problem of crucible fracture seems to have been satisfactorily solved, there has not been time to optimize the time-temperature cycles required to produce good optical quality glass from these crucibles, or to produce the high purity materials of the various core and cladding compositions required for the fabrication of an optimized clad laser rod.

### 3.4 EVALUATION OF CLAD PHOSPHATE LASER RODS

While awaiting delivery of the special crucibles an attempt was made to obtain some feeling for the level of performance that might be expected from a clad phosphate laser rod. Laser performance tests were run on rods of the purest materials then available and using a cavity configuration of the best rod and flashtube dimensions derived at that time.

The clad laser rod used in this test contained a core of MG-2399-M composition (0.2 wt%  $\text{Er}_2\text{O}_3$ , 15.0 wt%  $\text{Yb}_2\text{O}_3$  and 0.15 wt%  $\text{Nd}_2\text{O}_3$ ) and a cladding of MG-2419-A (3.0 wt%  $\text{Nd}_2\text{O}_3$  and 3.0 wt%  $\text{Yb}_2\text{O}_3$ ). This core glass was used in lieu of a composition containing just  $\text{Er}_2\text{O}_3$  and  $\text{Yb}_2\text{O}_3$  because this melt of glass was made from pure ingredients and was of good optical quality. It was hoped that the small amount of  $\text{Nd}_2\text{O}_3$  present would not cause significant quenching of excited  $\text{Er}^{3+}$ -ions. The beneficial role that this amount of  $\text{Nd}^{3+}$ -ions will play in the core of a clad rod configuration is uncertain. Presumably, much of the  $\text{Nd}^{3+}$  pump light will be absorbed by the higher  $\text{Nd}^{3+}$ -ion concentration in the cladding glass.



Laser rods 38, 50 and 76 mm long with a core diameter of 3 mm and a cladding diameter of 4 mm were tested with output mirror reflectivities ranging from 65 to 90%. The flashlamps were a nominal 4 mm diameter with a 3 mm bore and had an arc length equal to the laser rod length. Results of these measurements are given in Table VII. In all cases the inductance in the flashlamp circuit was 4460  $\mu$ H and the capacitance was 140  $\mu$ F.

TABLE VII. Clad Er-Phosphate Laser Rod Performance

Length (mm)	Output Reflectivity (%)	Laser Threshold (joules)	Slope Efficiency (%)	Input for 50 mJ Output (joules)
76	65	41	0.53	50
76	74.5	35	0.41	47
76	80	35	0.36	47
50	65	45	0.37	58
50	74.5	35	0.41	48
50	80	35	0.36	48
50	90	33	0.20	58
38	65	38	0.20	62
38	74.5	37	0.28	52
38	80	33	0.23	51
38	90	31	0.20	56

As may be seen in Table VII several combinations of rod length and output reflectivity yield laser outputs of 50 mJ for an input of 50 joules or less. By comparison, the best results achieved to date with silicate based glasses required an input of 90 joules for a 50 mJ output.\* This was obtained with a 38 mm long rod containing 0.3 wt%  $\text{Er}_2\text{O}_3$  (MG-2101 - MG-2400) with an output mirror reflectivity of 80%. (cf. Table II).

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\*Subsequent measurements on a clad silicate rod with an  $\text{Er}_2\text{O}_3$  concentration of 0.2 wt%, a core diameter of 3 mm and a length of 50 mm to test the findings of Section 2 have yielded an output of 50 mJ for an input of about 77 J in a cavity with an 80% output reflector using flashlamps with a 3 mm bore and 50 mm arc length feed from a 760  $\mu\text{F}$  condenser through a 4460  $\mu\text{H}$  inductor.

#### 4. SUMMARY AND CONCLUSIONS

A study of the effect of various clad laser rod parameters on laser performance and an attempt to develop a phosphate-based clad laser rod have been carried out in an attempt to achieve a 50 mJ single shot laser output with an input of 50 J.

The first attempt to fabricate a laser rod of an improved double-U configuration resulted in a rod with a distorted core and poor optical quality. The difficulty was associated with the starting materials which were chosen to allow a direct comparison with existing Er double-U laser rods. A second attempt was not made due to the limited effort available under the contract but the concept still appears to be a valid one.

The relative effects of  $\text{Er}^{3+}$ -ion content of the rod, rod length and active core diameter on laser performance have been investigated for silicate rods. It is anticipated that some compromise of these individual factors would be required for an optimized rod, likely values being 0.2 to 0.25 wt%  $\text{Er}_2\text{O}_3$ , 3 mm core diameter and 38 to 64 mm length. Subsequent to the contract effort, tests on a clad silicate rod with an  $\text{Er}_2\text{O}_3$  concentration of 0.2 wt%, a core diameter of 3 mm and a length of 50 mm produced a 50 mJ output at an input of 77 J.

Studies on additional sensitizing agents for phosphate materials suggest that MnO is the best candidate for additional sensitization of the clad phosphate rods. With the problems of melting high purity phosphate glasses solved, phosphate rods with MnO added to the cladding glass and possibly the core glass should be investigated.

Investigation of the compatibility and rod fabrication techniques of clad phosphate rods indicated that this phase of the program was feasible. The fabrication of good clad phosphate rods awaited the availability of good high purity phosphate core and cladding materials.

Late in the program a solution to the problem of making high purity phosphate materials without crucible failure was achieved. This came too late to fabricate and test optimized clad phosphate rods. In lieu of this and prior to the solution of the crucible problem, tests of clad phosphate rods of some of the best existing materials satisfied the project goal of a 50 mJ single shot output for a 50 J input.

With the various aspects of fabricating optimized clad phosphate rods now in hand, the engineering effort required to complete this work should be carried out to determine the ultimate efficiency of this laser system.



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